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Growth and Perfection of Silicon Crystals

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In order to investigate origins of defects such as dislocations and swirls, in situ observation of growth processes from the melt has been made by X-ray transmission topography, as shown schematically in Fig. 1. The appratus consisted of a high-power X-ray generator, topographic camera and imaging system. The X-ray generator with a Mo-rotating target was operated at 60 kV and 0.5 A with a focal size of 0.5 × 10 mm (Fig. 1a) The X-ray sensing camera unit and a specimen crystal are placed on the scan carriage of a Lang camera. By orienting the crystal so as to satisfy the Bragg condition for the slightly divergent X-ray incident beam ($K\alpha$), two images due to the diffracted $K\alpha_1$ and $K\alpha_2$ beams, each with a width of 1 mm, are received by the camera tube; two band-shaped regions of the crystal are imaged instantaneously on the picture monitor. Such images were recorded with a 16-mm cine camera synchronized with the TV scanning system and are called "direct-view images" for convenience. To display Lang topographs, the video signals due to the $K\alpha_1$ image are selected electrically and stored in the image storage unit, while the carriage is moved at a rate of 1 mm/sec. At the end of the carriage motion, a Lang topograph with the imaging area of the camera tube (9 × 13 mm) is displayed on the monitor. Hereafter, such images are referred to as "synthesized images".

To observe melting and growth processes, a furnace was mounted on the Lang camera. A Si crystal was placed between two carbon heaters, as shown schematically in Fig.1. The middle part of the crystal was melted in an argon gas flow. To observe growth processes, the floating zone method was applied by moving both the crystal and TV camera. Topographic images from the crystal were observed through the carbon heater by the TV camera.

The specimens were plate-shaped crystals with thicknesses of 0.2 to 0.3 mm which were cut from dislocation-free and swirl-free crystals. The orientation of the specimens is indicated in Fig. 2a.

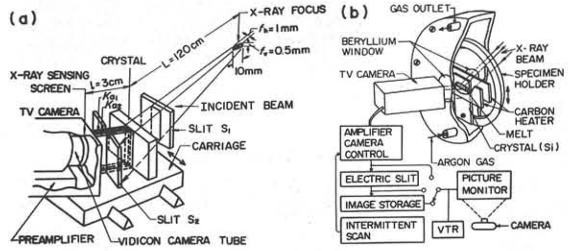

Fig. 1. In situ X-ray observation of melting and growth processes of Si crystals.

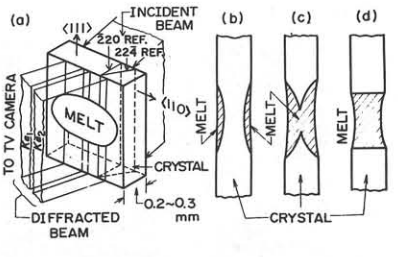

Fig. 2. Schematic illustration of the melting process. (b)-(d) Cross-section of the crystal in the melting sequences.

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MELTING PROCESS Different melting behavior was observed between dislocated and dislocation-free crystals: Just before melting, dislocated crystals becomes dislocation-free, and melting takes place from their surfaces. "Pendellösung fringes" (equal-thickness fringes) are observed due to wedge-shaped interfaces as shown in Fig. 2(c). This observation shows that crystals become highly perfect just before the melting.

On the other hand, droplets of the liquid are formed inside dislocation-free crystals, simultaneously with melting from their surfaces: Fig. 3(a) shows the state just before the melting. In Figs. 3(b) ∿ (e), there is an ellipse-shaped region which is covered with the melt. There seen many black spots. In Fig. 3(e), the crystal was very thin at the center of the ellipse, and many white holes appear by melting from the black spots. The spots were found to be due to droplets of the liquid silicon inside the crystal. It was found that microdefects are formed by solidification of the droplets and grow larger in the cooling process. It can be expected that such droplets, though small, are formed in the growth process of dislocation-free bulk crystals under the condition of remelt or temperature fluctuations. The droplets are considered to be an origin of microdefects "swirls".

GROWTH PROCESS An example is shown in Figs. 4(a) ∿ (f). The lower edges of each image is the interface between the crystal and melt, and the crystal is growing downwards. Fig. 4(a) is the state of the seed crystal just before the growth starts. The crystal is seen black due to many dislocations. In Fig. 4, a newly grown region is seen where many hairpin-shaped dislocations are inherited from the seed crystal, as shown schematically in Fig. 4(g). These dislocations follow the interface motion, touching the interface with their points [Figs. 4(b) and (c)]. In Fig. 4(e), however, the majority of the dislocations are left behind the interface. In Figs. 4(e) and (f), two dislocations intersect nearly perpendicular to the interface. They were found to be of composite type, each of which consists of three dislocations and is stable enough to intersect with the interface. As seen from the above observation, common dislocations cannot intersect with the growth interface, i.e., it was confirmed by some other observations that a dislocation-free region first grows and then dislocations moved in the newly grown region.

The results will be shown by a 16-mm movie film.

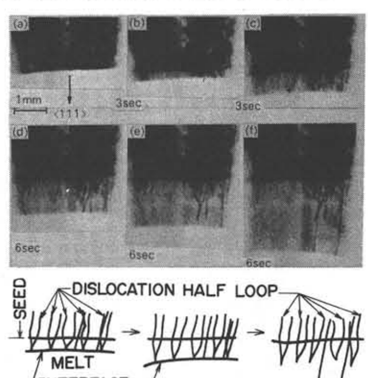

Fig. 4. Video topographs showing a grpwth process. Direct-view images Time intervals are indicated between the photographs.

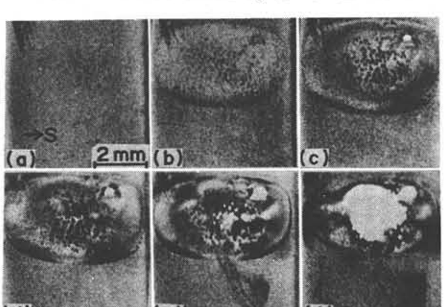

Fig. 3. Synthesized images showing the melting sequence of a dislocation-free crystal. 10 sec intervals.